# Deepwater Horizon – Data Validation Report Aliphatic Hydrocarbons by GC/FID

<b>SDG</b> : 1005014	Matrix: Water	Number of Samples: 15	
Laboratory: Alpha Analytical Services		Method/SOP: Alpha SOP/0-003 (Issue 6)	
Validation Level: Stage 2B Validation		Validation Criteria Table: MC252-AHC, Rev. 0	
Date of Report: July 239, 2010		Approved for Release:	

Refer to the SAMPLE INDEX for a list of validated samples.

Refer to the Data Validation Plan for validation approach, Criteria Tables, qualifier and reason code definitions.

The quality control (QC) elements that were reviewed are listed below.

1	Data Package Completeness		Sample Duplicate Analysis	
	√ Verification of EDD to Hardcopy Data Package		Blank Spike/Blank Spike Duplicate Sample Analyses	
1	1 Chain-of-Custody and Sample Receipt		Reference Material Analysis	
	$\sqrt{}$ Holding Times		Internal Standards	
	Retention Time Windows		Detection Limits	
	Initial Calibration		Target Analyte List	
	Initial Calibration Verification	2	Compound Quantitation	
	Continuing Calibration	1	Compound Identification (Stage 4 only)	
2	2 Method Blank Analysis		Mass Discrimination (Stage 4 only)	
	Surrogate Compound Recovery	1	Calculation Verification (Stage 4 only)	

<sup>√</sup> Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

#### **Overall Assessment**

Data were qualified as not detected or estimated based on method blank contamination. Data were also estimated based on BS/BSD precision outliers, and matrix interference.

All data, as qualified, are acceptable for use.

1005014 SHC Page 1 of 5 EcoChem, Inc.

<sup>1</sup> Quality control results are discussed below, but no data were qualified.

<sup>2</sup> Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed in this Data Validation Report.

#### **Data Package Completeness**

The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative. The laboratory sample receipt form was not present in the report for this SDG; however, the six coolers received were logged in to two SDG, 1005013 and 1005014. The sample receipt form was present in SDG 1005013 and no further action was taken. The laboratory submitted all other required deliverables.

# **Verification of EDD to Hardcopy Data Package**

Sample results and related quality control data were received in both an electronic and hardcopy format. Electronic data were verified against the laboratory report; no errors were found.

## **Chain-of-Custody and Sample Receipt**

Sample identification (ID) numbers listed on the chain-of-custody record are consistent with the sample ID reported in the laboratory electronic data deliverable (EDD) and hardcopy data package.

With the exceptions noted below, samples were received within the advisory temperature range of 2° to 6°C (Analytical Quality Assurance Plan (AQAP), Section 3.1). The laboratory noted all sample conditions on the sample receipt form.

The laboratory received several of the sample coolers with temperatures above the advisory control limits, ranging up to 9.9°C.

# **Holding Times**

Samples were analyzed within the holding time specified in the Analytical AQAP, Section 3.1, and documented in the Validation Criteria Table.

## **Initial Calibration**

Initial calibration (ICAL) standards were analyzed at the required frequency and the percent difference (%RSD) values were within the control limits specified in the AQAP, Table 6.1b and documented in the Validation Criteria Table.

#### **Initial Calibration Verification**

Initial calibration verification (ICV) standards were analyzed required frequency and the percent recovery (%R) values were within the control limits specified in the AQAP, Table 6.1b and documented in the Validation Criteria Table. The ICV was a separate standard prepared from a second source.

## **Continuing Calibration**

Continuing calibration (CCAL) standards were analyzed at the required frequency and the percent difference (%D) values were within the control limits specified in the AQAP, Table 6.1b and documented in the Validation Criteria Table.

#### **Method Blank Analysis**

To assess the impact of each blank contaminant on the reported sample results, two action levels are established at two (2x) and five times (5x) the concentration reported in the blank. If a contaminant is reported in an associated field sample and the concentration is less than the lower (2x) action level, the result is qualified as not detected (U-7). If a contaminant is reported in an associated field sample and the concentration is less than the higher (5x) action level, the result is qualified as estimated (J-7). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the higher action level, or for non-detected results.

The following table summarizes the method blank contamination and action levels:

Blank ID	Analyte	Popult (ug/L)	Action Limits (μg/L)	
DIATIK ID		Result (µg/L)	2x	5x
	n-dodecane (C12)	0.00103	0.00206	0.00515
	n-hexadecane (C16)	0.00168	0.00336	0.0084
	n-heptacosane (C27)	0.00412	0.00824	0.0206
SW051810B02	n-octacosane (C28)	0.00399	0.00798	0.01995
300001010002	n-nonacosane (C29)	0.00503	0.01006	0.02515
	n-triacontane (C30)	0.004614	0.00928	0.0232
	n-hentriacontane (C31)	0.00631	0.01262	0.03155
	Total Extractable Matter (C9-C44)	0.652	1.304	3.26

# **Surrogate Compound Recovery**

The percent recovery (%R) values for surrogates were within the control limits of 40% - 125%.

#### **Sample Duplicate Analysis**

Duplicate sample analysis was not performed for this sample batch. Precision was evaluated using the blank spike duplicate analysis.

## Blank Spike/Blank Spike Duplicate Sample Analyses

One set of blank spike/blank spike duplicate (BS/BSD) samples (for each analytical batch of 20 or fewer samples) was extracted and analyzed. The percent recovery (%R) and relative percent difference (RPD) values were calculated and evaluated.

The blank spike %R values for n-nonane (C9) and n-decane (C10) were less than the lower control limit. The BSD recoveries for these analytes were acceptable; therefore no action was taken.

The RPD values were less than the control limit of <30%, with the following exceptions:

Analyte	RPD
n-Nonane (C9)	91
n-Decane (C10)	83
n-Dodecane (C12)	48

The positive results for these compounds were estimated (J-9) in all associated samples.

## **Reference Material Analysis**

An aliquot of North Slope Crude was analyzed as a reference material. All recovery values were within the laboratory defined criteria of 65% - 135%.

#### **Internal Standards**

The percent recovery (%R) values for the internal standard (IS) were within the control limits of 50% - 200% of the area in the associated CCAL.

## **Compound Quantitation**

The laboratory applied a J-flag to all results between the quantitation limit (QL) and the method detection limit (MDL). During validation, results reported at less than the MDL were qualified as "found" (F).

The laboratory flagged the results for several analytes with a "G", indicating matrix interference that affected quantitation. These "G" flagged results were estimated (J-14).

#### **Mass Discrimination**

The ratio for the raw areas of n-C36 to n-C20 (calculated for the ICAL and CCAL) was  $\geq 0.70$ .

#### Calculation Verification

Stage 4 validation was performed on this SDG. No transcription or calculation errors were found.

# Attachment 1: Sample Index - SDG 1005014 Aliphatic Hydrocarbons by GC/FID

Sample ID	Lab ID	Date Collected	
JF.4KM.DEEP.WD.20100512.N088	1005014-01	5/12/2010	
JF.8KM.BLANK.DIWD,20100512.N075	1005014-02	5/12/2010	
JF.2KM.MIX75.WD.20100513.N136	1005014-03	5/13/2010	
JF.2KM.MIX30.WD.20100513.N140	1005014-04	5/13/2010	
JF.2KM.400.WD.DUP.20100513.N160	1005014-05	5/13/2010	
JF.2KM.DEEP.WD.20100513.N124	1005014-06	5/13/2010	
JF.2KM.SURF.WD.20100513.N148	1005014-07	5/13/2010	
JF.8KM.MID.WD.20100512.N051	1005014-08	5/12/2010	
JF.8KM.SURF.WD.20100512.N071	1005014-09	5/12/2010	
JF.8KM.MIX30.WD.20100512.N067	1005014-10	5/12/2010	
JF.4KM.SURF.WD.20100512.N112	1005014-11	5/12/2010	
JF.2KM.LEESURF.WD.20100513.N168	1005014-12	5/13/2010	
JF.2KM.135FT.WD.20100513.N132	1005014-13	5/13/2010	
JF.2KM.BLANK.DIWD.20100513.N164	1005014-14	5/13/2010	
JF.2KM.400.WD.20100513.N152	1005014-15	5/13/2010	

1005014\_SHC Page 5 of 5 EcoChem, Inc.